

AD-A245 836



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CONTRACTOR REPORT BRL-CR-680

BRL

LIQUID GUN PROPELLANT (LGP)
1846 PILOT PRODUCTION PROGRAM:
FINAL REPORT

R. G. BRASFIELD
R. A. BIDDLE
J. MALLICK
THIOKOL CORPORATION

JANUARY 1992



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REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE January 1992		3. REPORT TYPE AND DATES COVERED Final, May 87-Jan 91
4. TITLE AND SUBTITLE Liquid Gun Propellant (LGP) 1846 Pilot Production Program - Final Report			5. FUNDING NUMBERS C:DAAD05-87-C-8030	
6. AUTHOR(S) R. G. Brasfield, R. A. Biddle, and J. Mallick				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Thiokol Corporation, Elkton Division Elkton, MD 21921			8. PERFORMING ORGANIZATION REPORT NUMBER E89-91	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Ballistic Research Laboratory ATTN: SLCBR-DD-T Aberdeen Proving Ground, MD 21005-5066			10. SPONSORING / MONITORING AGENCY REPORT NUMBER BRL-CR-680	
11. SUPPLEMENTARY NOTES The Contracting Officer's Representative for this report is Charles S. Leveritt, U.S. Army Ballistic Research Laboratory, ATTN: SLCBR-IB-B, Aberdeen Proving Ground, Aberdeen, MD, 21005-5066.				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) The Liquid Gun Propellant (LGP) 1846 Pilot Production program was begun in May 1987 to support the Army LP gun development effort at General Electric. More than 18,000 kg of LGP were produced at the Elkton Division of Thiokol Corporation and delivered in support of the testing program at Wright-Malta, Ballston Spa, NY. The LGP was produced at the Elkton plant facilities utilizing one 50-gallon and one 500-gallon glass-lined temperature-controlled reactor. Dilute hydroxyl ammonium nitrate (HAN) was concentrated to 80-85% by weight. Triethanol ammonium nitrate (TEAN) was synthesized from triethanolamine (TEA) and nitric acid. Purity of the product was such that recrystallization was not required. Packaging and shipment were accomplished using DoT 2S/6D double-pack containers filled to 25 kg (~ 5 gal) and packed within DoT 17H metal drums. Shipping classification of explosive liquid class B was utilized per an Interim Hazard Classification furnished by Ballistic Research Laboratory, Aberdeen Proving Ground, MD (BRL/APG).				
14. SUBJECT TERMS Liquid Propellants; LP; Liquid Gun Propellants; LGP; LP 1846; LP 1845; LGP 1846; LGP 1845; Hydroxylammonium Nitrate; HAN; Triethanolammonium Nitrate; TEAN; Triethanolamine; TEA; Production			15. NUMBER OF PAGES 74	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT SAR	

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TABLE OF CONTENTS

	<u>Page</u>
LIST OF FIGURES	v
LIST OF TABLES	v
1. INTRODUCTION	1
1.1 Program Requirements	1
1.2 Schedule	2
1.3 Shipping and Delivery	2
2. PRODUCTION PROCESSES	2
2.1 B-1 Pilot Production Plant	2
2.2 Raw Materials and Requirements	3
2.3 Manufacturing Procedures	6
2.3.1 HAN Concentration	6
2.3.2 TEAN Synthesis	7
2.3.3 Blending of LGP1846	7
2.4 Packaging of LGP1846	8
2.4.1 Packaging Component Preparation	8
2.4.2 Filling the LGP Interior Package	8
2.4.3 Packaging for Shipment	8
2.5 Material Safety Data Sheet (MSDS) and Interim Hazard Shipping Classification (HSC)	10
3. DELIVERABLES	10
3.1 Destination	10
4. QUALITY ANALYSES AND PROCEDURES	10
4.1 Receiving Acceptance of Raw Materials and Components	10
4.2 In-Process and Blended LGP Analyses	12
4.3 Analytical Procedures	12
5. CONTRACT STATUS AND PERFORMANCE	14
6. PROGRAM SUPPORT	16
APPENDIX A: STATEMENT OF WORK	17
APPENDIX B: SHIPPING ARRANGEMENT DRAWING	23
APPENDIX C: MSDS/HSC FORM	27

	<u>Page</u>
APPENDIX D: REPRESENTATIVE QUALITY LAB DATA	43
APPENDIX E: ANALYTICAL PROCEDURES ANALYSIS WORKSHEETS PRODUCT RELEASE FORMS	53
DISTRIBUTION LIST	69

LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
1. Reactor Jacket Temperature Controlled by Primary Cascade	4
2. Reactor Jacket Temperature Controlled by Secondary Cascade	5
3. General Process Scheme	6
4. Combined Acid/Oxime Titration Curves	12
5. Excess Acid Titration in LGP	13

LIST OF TABLES

<u>Table</u>	<u>Page</u>
1. LGP Correction Analysis	9
2. Deliverables	11
3. Lot Data Summary of LGP	13
4. Line Items	14
5. Contract Amendments	15



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1. INTRODUCTION

This report summarizes the work accomplished in support of contract DAAD05-87-C-8030 for the manufacture and delivery of Liquid Gun Propellant (LGP) 1846. The basis for this program originated with our proposal EP572-86, dated September 17, 1986, in response to the Ballistic Research Laboratory, Aberdeen Proving Ground, MD (BRL/APG), solicitation DAADO5-86-R-8334, dated August 15, 1986.

A total of 18,050 kg of LGP was produced and delivered under the contract, from May 1987 to January 1991. The program team at Thiokol/Elkton included Richard Biddle, research chemist; Joseph Lehman, analytical chemist; John Mallick, process engineer; Warren Brownell, quality engineer; Shirley Bonsell, quality control chemist; Herman Mitchell, contract administrator; Dorothy Ryan, publications; and Ray Brasfield, program manager. Technical representation at BRL were Mr. Charles Leveritt and Ms. Josephine Wojciechowski.

1.1 Program Requirements. The program requirements include the technical statement of work, schedule of deliverables, and the packaging and shipping details.

- Statement of Work (SOW). The SOW included the following principal areas. The complete SOW is contained in Appendix A for reference.

The end item furnished under this contract is LGP1846. The formulation for LGP1846 is 60.8% hydroxyl ammonium nitrate (HAN), 19.2% triethanol ammonium nitrate (TEAN), and 20.0% water. A tolerance of $\pm 0.5\%$ for these percentages is allowed.

The HAN must be concentrated from dilute aqueous solutions. This must be done by stripping water off under vacuum at a temperature not to exceed 55° C. The only domestic supplier of aqueous HAN is Southwest Analytical Chemicals (SACChem) of Austin, TX. The SAC HAN is 24–26% by weight.

The TEAN must be synthesized from triethanol amine (TEA) and nitric acid. The TEA must be of the highest quality and 97% pure minimum. Thiokol utilizes fresh 99% TEA in its production.

The nitric acid must be reagent grade. Thiokol procures 70% by weight nitric acid for TEAN synthesis. The amine is added to the dilute acid in a reactor controlled to maintain temperature at less than 4° C. Because of the resultant purity and high quality level of the raw materials, Thiokol has not been required to recrystallize the TEAN product to remove contamination.

The product analyses include metal contamination (iron, nickel, copper, lead, and tin), UV analysis for total nitrate (285–315 nm), and titration for HAN, TEAN, and total nitrate content.

1.2 Schedule. The schedule of performance for this program included the period from May 1987 through January 1991. Initial contract release was followed by procurement of raw materials and shipping hardware. The production status and substance schedule were updated with each monthly report. Deliveries of LGP were begun in the fall of 1987 and the final delivery was made in January 1991.

1.3 Shipping and Delivery. The shipping arrangement is defined by Thiokol drawing E40378. This arrangement consists of a DoT 2S polyethylene container of approximately 19L volume with a DoT 6D metal overpack. This nested container arrangement is filled with 25 kg of LGP. The fill port is fitted with a 15 psig pressure relief valve and is covered with a tamperproof witness tape for security purposes. This entire package is placed within a 114L DoT 17H open-head steel drum and packed with vermiculite on all sides. This 17H steel drum functions as the outer shipping container for transportation of the LGP. The shipping arrangement drawing is presented in Appendix B.

2. PRODUCTION PROCESSES

2.1 B-1 Pilot Production Plant. The B-1 pilot production plant consists of a Pfaudler 500-gallon glass-lined, insulated steel reactor. This unit is coupled to a temperature-control system and a steam jet ejector vacuum system. It is fitted with a D baffle and stirrer. This plant also contains a Pfaudler 50-gallon glass-lined, insulated steel reactor. This unit is also coupled to a temperature-control system and is fitted with a similar D baffle and stirrer. Both reactors are controlled by a Leeds and Northrop (L&N) primary and secondary cascade programmable remote control system.

- **Description of Control System.** The L&N process management equipment is utilized to control the concentration of HAN, the production of TEAN, and the mixing of LGP.

During early work on the concentration of HAN, the temperature of the reactor jacket was controlled by primary cascade; that is, by the equipment that controls the reactor jacket temperature in response to the temperature of the product (HAN). This was found to give frequent oscillations in the reactor jacket temperature. Subsequently, it was found that by utilizing secondary cascade or controlling the reactor jacket temperature directly, the temperature oscillations in the reactor jacket were eliminated and the process was more efficient (Figures 1 and 2). During the HAN concentration, the reactor jacket inlet and outlet temperatures and the product temperature are recorded for subsequent analysis. These temperature data along with other process information such as the reactor vacuum level are recorded in digital form and printed at 5-minute intervals.

During TEAN production, the addition and monitoring of the TEA to the 50-gallon reactor is controlled with the L&N processing management system. The product temperature and the reactor inlet and outlet temperatures are monitored and recorded as outlined for the HAN concentration described above. The chiller for this reactor is set at a desired temperature and is controlled by the chiller control system and not with the L&N system.

Mixing of the LGP is accomplished in the 500-gallon reactor. Temperature control is not needed during this operation.

In summary, all temperature monitoring and recording is completed with the L&N processing management system. All heating control in both the 50- and 500-gallon reactors is accomplished with the L&N system. The cooling control for both reactors is accomplished with the cooling units and is independent of the L&N system except for the temperature monitoring and recording. The starting and stopping of the reactor agitators and the TEA pump is accomplished through the L&N system. The equipment control is not completely automated and is part electronic and part manual.

2.2 Raw Materials and Requirements. The production LGP1846 (or 1845) requires only three basic raw materials, plus water. Hydroxyly ammonium nitrate (HAN) is purchased in a

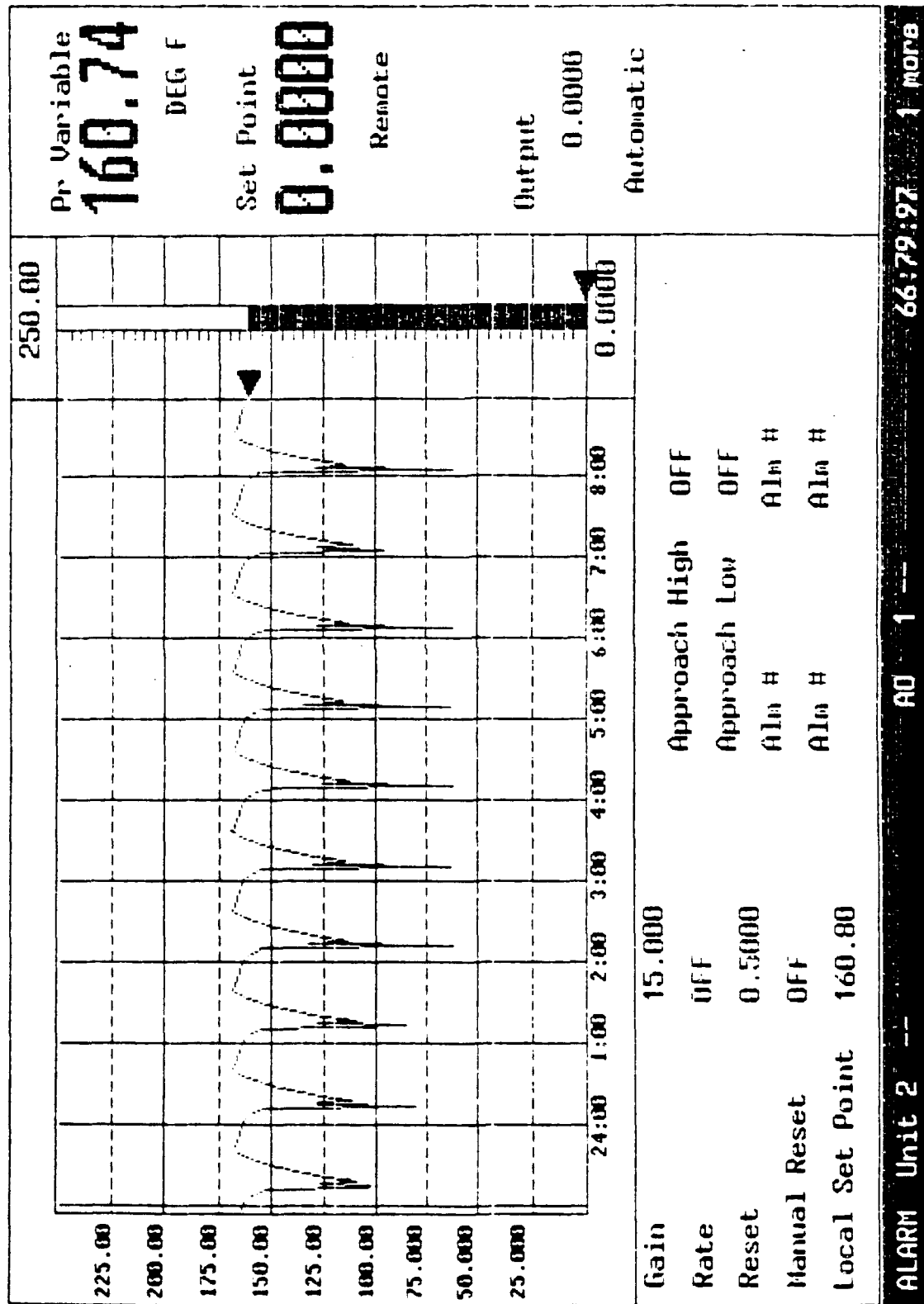


Figure 1. Reactor Jacket Temperature Controlled by Primary Cascade.

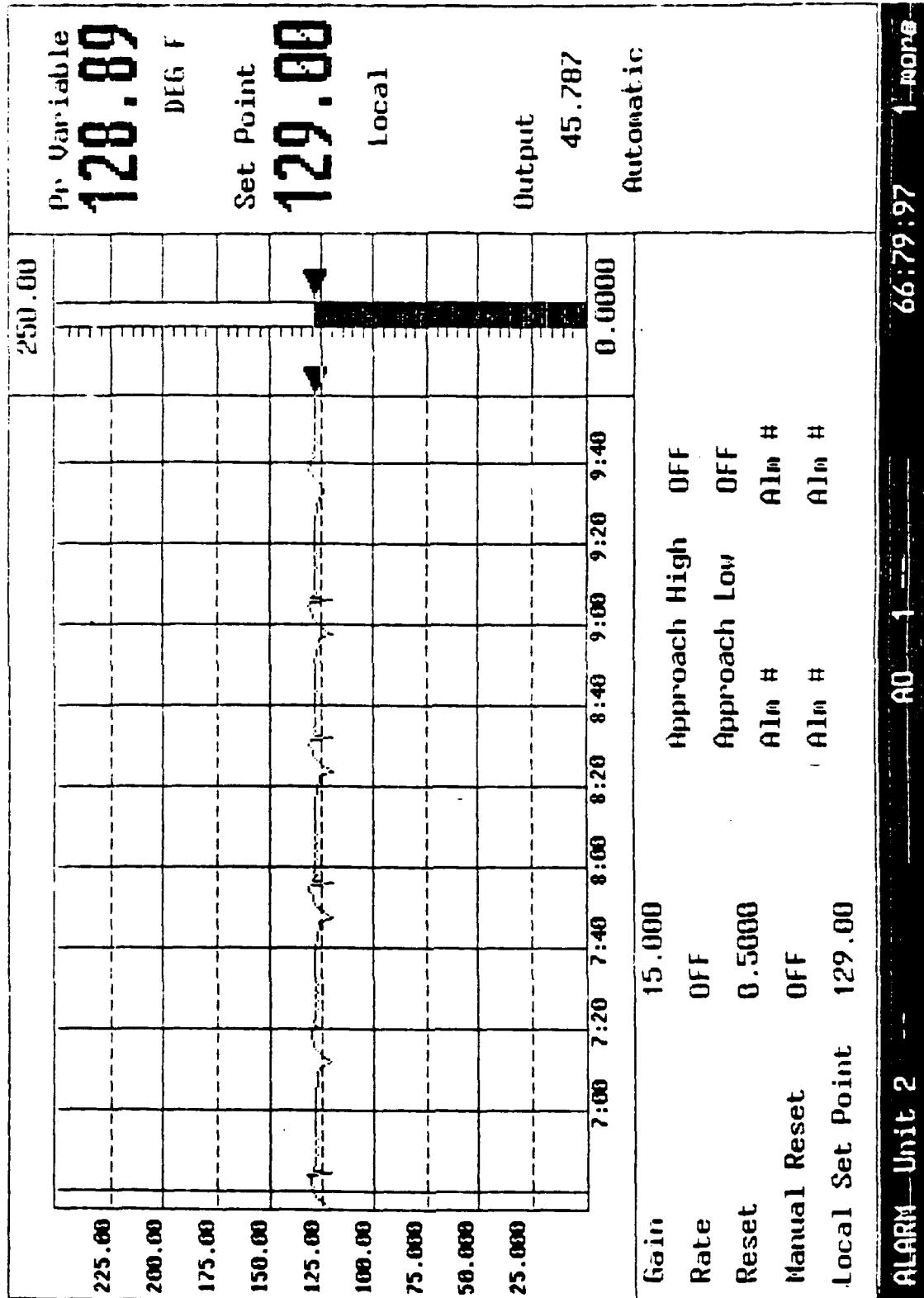


Figure 2. Reactor Jacket Temperature Controlled by Secondary Cascade.

dilute aqueous solution (24–26% by weight) from the only current domestic supplier SACHem of Austin, TX. Triethanol amine (TEA) is purchased at the highest purity (99%) commercially available. Because it is produced for the cosmetics industry in tremendous quantity, availability and delivery are readily assured. The important processing aspect for TEAN synthesis is TEA purity and freshness. We have found that fresh, 99% pure TEA produces the highest quality TEAN. Nitric acid, reagent grade, is purchased at 70% by weight and obtained through normal commercial distributors.

2.3 Manufacturing Procedures.

2.3.1 HAN Concentration. The flow diagram for the manufacturing process is shown in Figure 3. The dilute HAN (24–26% by weight) must be concentrated prior to blending for LGP production. This concentration is accomplished by a simple stripping of water under vacuum with controlled heating of the HAN. As-received HAN is transferred into the 500-gallon Pfaudler reactor, with an initial loading of five 55-gallon barrels. The data collection instruments are turned on, the stirrer operated, and the reactor jacket heating begun. Temperature is controlled to $50 \pm 5^\circ \text{C}$. Vacuum is obtained by the steam jet ejector system

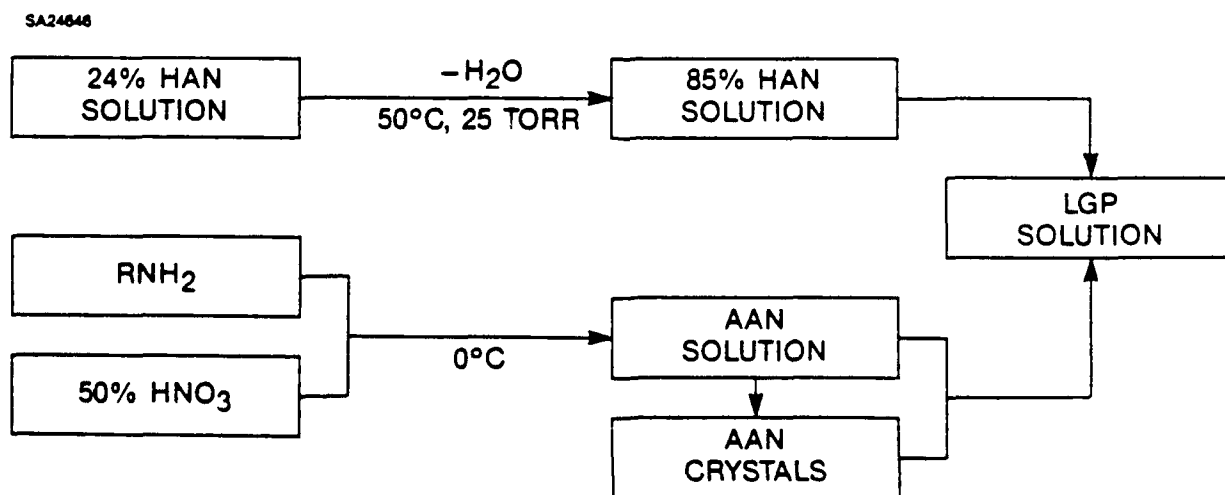


Figure 3. General Process Scheme.

and this is activated last. A target vacuum of less than 23 mm Hg is obtainable with this system. After 24 hours, the vacuum is released and another barrel of dilute HAN is transferred into the reactor, the reactor resealed, and the vacuum reestablished. This process continues until a total of 12-drums have been concentrated. The target concentration is 80–85% and takes 9–11 days for the 12-drum lot. The reactor vacuum and temperature are allowed to return to ambient conditions before samples are thieved for analysis. The concentrated solution is drained from the reactor and stored in the original HAN solution drums (55-gal) for LGP preparation.

2.3.2 TEAN Synthesis. Triethanol ammonium nitrate (TEAN) must be produced from the reaction of TEA with nitric acid. This reaction produces a large heat of neutralization; therefore, the reactor must be cooled constantly during synthesis. The as-received nitric acid is added to the 50-gallon Pfaudler reactor, with an initial loading of 84 ± 1 lb. The acid is chilled to -10°C and 29 ± 1 lb of demineralized water is added, while continuing to maintain the reactor contents temperature of -10°C . The reactor contents are also constantly stirred during the synthesis. When the reactor temperature has stabilized, the as-received TEA is slowly added into the reactor at a rate not exceeding 5 lb per hour. TEA is added at a controlled rate, in conjunction with reactor temperature control system, so that the heat of reaction does not outpace the system capability to maintain temperature control at -10°C . Synthesis is completed when a dilute sample ($\times 15$) gives a pH of 6.3. This is at approximately 139 lb TEA for the reactor loading of 84 lb nitric acid. Synthesis time for an average lot is about 60 hours and produces 110–120 lb TEAN from a centrifuge in a damp state with less than 5% retained water. This material is immediately redissolved in demineralized water to prepare a nominal 80% by weight TEAN solution and stored for LGP preparation.

2.3.3 Blending of LGP1846. Blending of the LGP takes place in the 500-gallon Pfaudler reactor. The total weight of approved HAN and TEAN that is available for blending determines the quantity of LGP that can be prepared. Only materials that have been analyzed and given a written release from the Quality Lab are used in the final LGP blending operation. The calculated weights of HAN, TEAN, and water are added to the reactor and stirred at ambient temperature for 2 hours. Corrections are made to the blend as indicated by

the batch analysis until the correct LGP1846 formulation is achieved. See Table 1 for correction algorithm.

2.4 Packaging of LGP1846.

2.4.1 Packaging Component Preparation. Packaging of the LGP requires a preparation step prior to filling the containers. The 2S polyethylene container as purchased is made from virgin (unrecycled) material. To preclude any opportunity for plasticizers, or other fabrication aids, and particulate matter from contaminating the LGP, the 2S polyethylene containers are rinsed vigorously five times with deionized water. Then the containers are filled again with deionized water and leached at 150° F for 48 hours minimum. After drying, these prepared containers are then bagged with clear polyethylene film and tagged as clean, approved for use in packaging LGP.

2.4.2 Filling the LGP Interior Package. After the Quality Lab has analyzed and released the LGP lot for packaging, the solution can be drained from the reactor. Clean, 5-gallon 2S polyethylene containers, approved for use, are tare weighed and prepared for filling. A small volume, approximately 2–5 gallons, of LGP is drained through the bottom reactor valve to clear it. Each 2S container is then filled by weight to 25 kg. Three packaging samples of LGP (early, middle, and end stages of the filling operation) are taken and sent to the laboratory for metals analysis by atomic absorption (AA), total nitrate UV trace, and free acid analysis. After filling, each 2S container is fitted with a 15-psi valve closure and a tamperproof witness tape.

2.4.3 Packaging for Shipment. The final packaging arrangement is shown in Thiokol drawing E40378. The filled 2S containers are placed within a 6D metal overpack container with a crimp-on lid. This double-pack container is then placed inside a 3-mil (minimum) thickness polyethylene bag, the air expelled, and the bag taped closed.

The outer shipping container is a DoT 17H, 18-gauge steel drum with an open-head-style lid. A 30-gallon size drum is used, with grade 4 vermiculite (exploded mica) as the packaging dunnage. The 17H drum is filled to approximately 6 inches with vermiculite, and the completed double pack is centered inside. Additional vermiculite is added all around until the

Table 1. LGP Correction Analysis

1. Record Weight of LGP in Reactor _____

Spec. Allowable

2. Record Lab Analysis	HAN	_____	0.608 ± 0.005
	TEAN	_____	0.192 ± 0.005
	H ₂ O	_____	0.200 ± 0.005
			1.000

		<u>Reactor Weight</u>		<u>Lab Analysis</u>		<u>Weight</u>
3. Analysis	HAN	= _____	X (HAN)	_____	= _____	
Weights	TEAN	= _____	X (TEAN)	_____	= _____	
	H ₂ O	= _____	X (H ₂ O)	_____	= _____	

4. A. Determine which item is high out specification. If only one item, try TEAN first then HAN.

Take Item Weight from Lab Analysis and Divide

Circle Item 1	HAN	_____	x	_____	÷	_____	=	_____
	TEAN	LGP Weight		Lab Analysis		Item Spec		Revised LGP
	H ₂ O			Item		(Allowable Midpoint)		Weight Total

B. Circle Item 2	HAN	_____	x	_____	=	_____
	TEAN	Revised		Second Item		Total Weight of
	H ₂ O	LGP Weight		Spec. Allowable		Item Required

_____	-	_____	=	_____
Total Weight of		Item Weight From		Weight of Item
Item Required		3 Above		Required

5. Circle	HAN	_____	+	_____	=	_____
	TEAN	Weight of Item 4B		% Concentration		Solution Weight of
	H ₂ O	Required		of Material + 100		Item 4B Required
				(Available)		

6. A. Presumed Water	_____	+	_____	=	_____
	Revised Weight of		Spec. Allowable		Weight of H ₂ O Required
	LGP from 4A		H ₂ O Midpoint		

B.	_____	-	_____	-	_____	=	_____
	Weight of Water		H ₂ O Weight From 3		Solution Weight		Additional
	Required 6A				From Item 5 x		Water Required
					(H ₂ O % + 100)		

17H drum is filled. The lid is then put in place and the attachment ring plus its securing bolt are damped to seal the drum.

Labels and stenciling are per the details on drawing E40378 and the destination address per the contract line item.

2.5 Material Safety Data Sheet (MSDS) and Interim Hazard Shipping Classification (IHSC). The current MSDS for the LGP1846 is shown in Appendix C. The IHSC authorizing shipment of this material was obtained from BRL and a copy is also shown in Appendix C.

3. DELIVERABLES

Contract DAAD05-87-C-8030, as amended, specified the delivery of 18,050 kg of LGP: 16,450 kg of LGP1846 and 1,600 kg of LGP1845, which were delivered to the U.S. Army from October 1987 to January 1991.

3.1 Destination. Shipments were made to the following destinations (Table 2):

- a) BRL-Aberdeen Proving Ground, MD
- b) General Electric, c/o Wright-Malta, Balston Spa, NY
- c) Letterkenny Arsenal, Chambersburg, PA
- d) Geo Centers, Inc., Wharton, NJ

4. QUALITY ANALYSES AND PROCEDURES.

The quality procedures for this program varied from that of certification of conformance to published requirements (for some raw materials and components) to detailed chemical analyses of the finished LGP product.

4.1 Receiving Acceptance of Raw Materials and Components. The hydroxyl ammonium nitrate (HAN), triethanol amine (TEA), and nitric acid were the raw materials purchased for this program and each was received under different criteria. The nitric acid, purchased as reagent grade material, was accepted on a certificate of conformance from the supplier. The TEA was

Table 2. Deliverables

LGP1846			
Lot	Qty, kg	Date	Destination
-01	500	Oct 87	GE/BRL/GEO
-02	1,675	Dec 87	GE
-02, -03	1,675	Jan 88	GE
-04	1,650	Mar 88	GE/BRL
-03, 04, 05	2,050	May 89	GE/BRL
05, 06	2,050	Jun 89	Letterkenny/BLR
-06, 07	2,075	Jul 89	Letterkenny/BRL
-07, 08	2,075	Aug 89	Letterkenny/BRL
09	1,375*	Jan 90	GE/BRL
-08, 09, 10, 11	800	Jun 90	GE/BRL
-11	300	Jan 91	GE
LGP1845			
-01	1,600	May 88	Letterkenny/BRL

*From GFM concentrated HAN

also accepted with a certification of conformance but to a >99% purity requirement and lot date. The HAN was analyzed by the Quality Laboratory initially to establish the acceptability and reproducibility of the commercially produced material. Following evidence of reproducible quality (test data from the first few deliveries), the HAN was accepted to the certification of conformance and lot data supplied by the vendor. Reanalysis was done any time the vendor production cycle was broken and needed to be restarted, essentially a "first-article" checkout of the production. Data obtained for the HAN receiving analysis were percent HAN, trace metal contamination, and free acid analysis.

An amendment to the contract provided Government-furnished concentrated HAN (from NOS/IH) for production of additional LPG1846. The receiving acceptance of this GFM was approved only after a complete nitrate, water, and trace metals analysis. Approximately 2,000 lb of GFM was received.

4.2 In-Process and Blended LGP Analyses. In-process analyses followed the production acceptance of TEAN synthesis and HAN concentration, prior to blending. In-process TEAN analysis included titration for TEAN content, Karl Fischer analysis for water, and UV analysis for total nitrate (optional). In-process HAN analysis included titration for HAN content, Karl Fischer analysis for water, and AA for iron contamination.

Blended LGP analyses included titration with aqueous sodium hydroxide using the double break point technique for HAN and TEAN content (Figure 4), total nitrate by UV, water content by Karl Fischer analysis, trace metal analysis by AA, and free acid content using titration with sodium hydroxide (Figure 5). Table 3 summarizes the LGP data for LGP1846 lots delivered under this program.

4.3 Analytical Procedures. The procedures for HAN, TEAN, LGP, metals, and free acid analyses have been developed over several programs in conjunction with BRL and other researchers. The procedural details and calculation/data sheets utilized for this program are presented in Appendix D for reference.

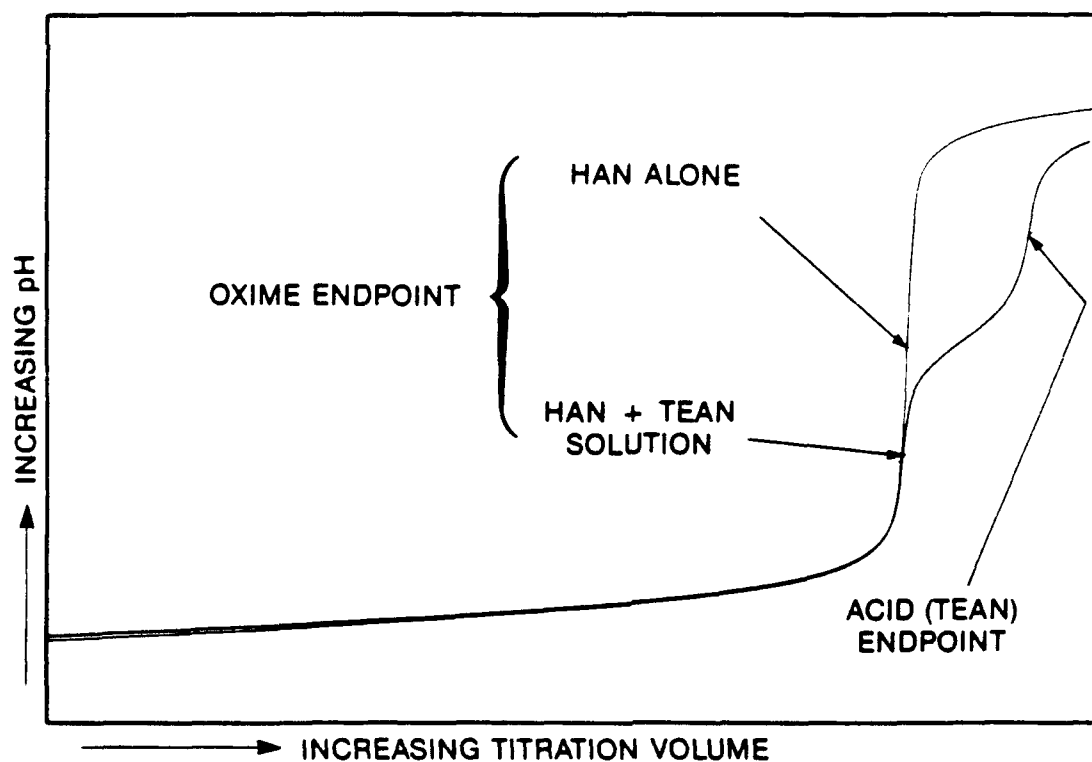


Figure 4. Combined Acid/Oxime Titration Curves.

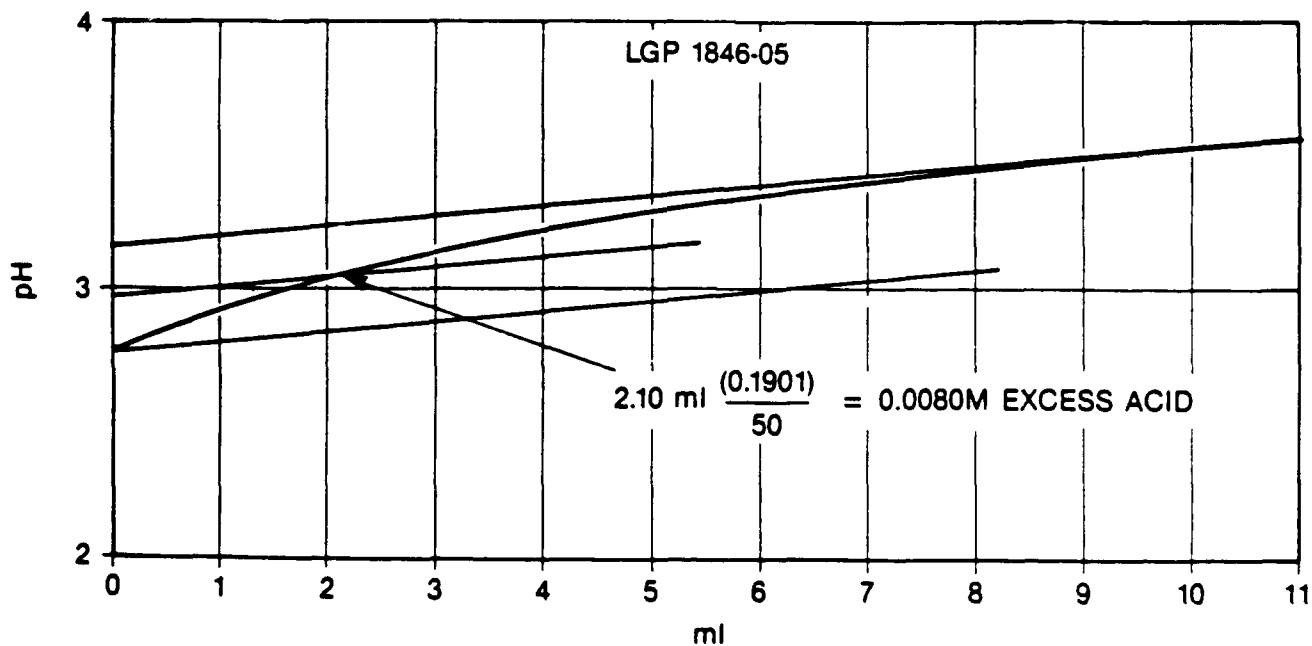


Figure 5. Excess Acid Titration in LGP.

Table 3. Lot Data Summary of LGP

LGP1845					
Lot	H ₂ O, %	HAN, %	TEAN, %	HAN/TEAN	Fe, ppm
-01	19.8	60.3	19.9	3.03	3
-02	20.3	61.1	19.1	3.20	1
-03	19.7	61.0	19.6	3.11	0.8
-04	20.2	61.1	19.4	3.16	0.7
-05	20.5	60.9	19.2	3.17	1.0
-06	19.6	61.0	19.5	3.13	1.4
-07	20.0	60.7	19.7	3.08	1.4
-08	19.6	60.7	19.6	3.11	1.6
-09	20.4	61.1	19.3	3.17	2.0
-10	19.7	61.0	19.4	3.14	1.4
-11	19.9	60.3	19.6	3.08	1.5
LGP1845					
-01	16.8	63.6	20.0	3.18	1.5
Requirements					
LGP1846	19.5 - 20.5	60.3 - 61.3	18.7 - 19.7	3.06 - 3.28	<5
LGP1845	16.3 - 17.3	62.7 - 63.7	19.5 - 20.5	3.06 - 3.28	<5

5. CONTRACT STATUS AND PERFORMANCE

The following tables (Tables 4 and 5) summarize the line items, amendments, and delivery performance for the contract.

Table 4. Line Items

CLIN	Description	Qty, kg	Contract Delivery	Actual Delivery
	Basic Year			
0001AA	Lot 1	500	Oct 14, 1987	Oct 27, 1987
0001AB	Lot 2	1,675	Dec 16, 1987	Dec 7, 1987
0001AC	Lot 3	1,675	Feb 24, 1988	Jan 15, 1988
	Option Year			
0002AA	Lot 1	1,650	Apr 30, 1988	Mar 11, 1988
0002AB	Lot 2	1,586 (LGP1845)	May 30, 1988	Apr 28, 1988
0002AC	Lot 3	2,050	May 30, 1989	May 8, 1989
0002AD	Lot 4	2,050	Jun 30, 1989	Jun 1, 1989
0002AE	Lot 5	2,075	July 30, 1989	July 7, 1989
0002AF	Lot 6	2,075	Aug 30, 1989	Aug 18, 1989
0005	GFM HAN Lot	1,600	Feb 15, 1990	Jan 5, 1990
0006	LGP1846	800	Jun 30, 1990	Jun 25, 1990
0001	LGP1846	300	Jan 10, 1991	Jan 18, 1991

Table 5. Contract Amendments

Amendment	Description	Date	CLIN Effectivity
P00001	Type error. Delete C,3.1. Delete I. 15.	May 1987	0001AB -- --
P00002	Change delivery from Sep 13 to 14, 1987.	Sep 1987	0001AA
P00003	Partial release op yr 1.	Sep 1987	0002AA
P00004	Add shipping charges. Change delivery destination.	Oct 1987	00004 0001AA
P00005	Partial release op yr 1. Change delivery from Oct 30, 1987, to Dec 16, 1987 Change delivery from Nov 30, 1987, to Feb 24, 1988.	Dec 1987	0002AA, AB 0001AB 0001AC
P00006	HAN price delta. Add water analysis by KF and double-break titration with aqueous NaOH for HAN and TEAN.	Feb 1988	-- --
P00007	Change delivery destination.	May 1988	0002AA
P00008	Change 1846 to 1845. Change delivery date from May 30, 1988, to June 30, 1988. Change destination.	May 1988	0002AB 0002AB
P00009	Change funding for shipping.	Sep 1988	0004
P00010	Funds, balance of option year, and delivery schedule. Change destination.	Jan 1989	0002AC,D,E,F 0002AC,D,E,F
P00011	Change destination.	Mar 1989	0002AC
P00012	Increase production of 1846 by 1,600 kg using GFM HAN.	Dec 1989	0005
P00013	Increase production of 1846 by 800 kg.	Jun 1990	0006
P.O. DAAD05-91-P-0968	Increase production of 1846 by 300 kg.	Dec 1990	0001

6. PROGRAM SUPPORT

During the LGP pilot production program, technical and management support was provided through program meetings, technical interchange meetings, participation in BRL-sponsored technical conferences, and participation in fact-finding and planning meetings with ARDEC and FDR personnel.

<u>Date</u>	<u>Meeting</u>	<u>Subject</u>
Jul 1987	Thiokol/BRL	LGP Fact-Finding and Planning
Aug 1987	Conference	3rd HAN-Based Liquid Propellant Conference
Dec 1987	Thiokol/BRL	Propellant Conference
Feb 1988	Thiokol/BRL/JPL	TIM on Analysis Procedures LGP Fact-Finding and Planning
Apr 1988	Thiokol/BRL/FDR	LGP Fact-Finding and Planning
Aug 1988	Conference	4th HAN-Based Liquid Propellant Conference
Jan 1989	Thiokol/ARDEC	HAN and LGP Scaleup Production and Packaging Logistics
May 1989	Thiokol/BRL/ARDEC/GEO	TIM on Free Acid Analysis
Aug 1989	Conference	5th HAN-Based Liquid Propellant Conference

APPENDIX A:
STATEMENT OF WORK

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PART I - THE SCHEDULE

SECTION B - SUPPLIES/SERVICES AND PRICES

C.3 The Contractor shall perform as set forth in Section B and described in C.4 below for each particular contract line item number (CLIN) .

C.3.1 The Contractor shall furnish the quantities as perscribed in Section B of CLIN 1000AA for First Article Government Testing in accordance with the provision in Section I at I.15 prior to continuance of remaining requirement.

C.4 The Contractor shall furnish the required chemicals to the Government at Aberdeen Proving Ground, Md in accordance with the requirements and specifications listed below.

C.4.1 Principal Features

C.4.1.1 The contractor shall furnish a total of 15,400 Kilograms (in accordance with Section B) of liquid propellant 1846. The liquid propellant 1846 contains 60.8% hydroxylammonium nitrate (HAN) 19.2% triethanolammonium nitrate (TEAN), 20.0% water.

C.4.1.2 The required HAN solution must be prepared from dilute solutions. HAN in solution at a concentration of from 18% to 24% (by weight) is commercially available. The straight forward way of preparing the concentrated solution is by stripping off the water under vacuum at a temperature not above 55°C. (131°F)

C.4.1.3 The only known supplier of dilute HAN in the United States is Southwest Analytical Chemicals, Austin, TX.

C.4.1.4 TEAN is not available commercially, and must be prepared by the vendor from the corresponding aliphatic amine and nitric acid. The vendor may choose his own supplier of the aliphatic amine. It must be at least 97.0% pure, as determined by gas chromatography.

C.4.1.5 Reagent-grade nitric acid must be used.

C.4.1.6 Manufacturing Guidelines for making TEAN:

- a. the amine must be added to the nitric acid, never the reverse.
- b. The temperature of the reaction vessel must not be allowed to rise above 40°C (100°F) at any time.
- c. The product must be recrystallized at least once from water.
- d. In removing the excess water, the temperature of the TEAN-water mixture must not be allowed to rise above 25°C (77°F).

PART I - THE SCHEDULE

SECTION B - SUPPLIES/SERVICES AND PRICES

C.4.1.7 Specifications:

a. The final products when shipped must not contain more than 5 parts per million (ppm) of any heavy metals, including (but not limited to) iron, nickel, copper, lead, and tin.

b. The composition of the liquid propellant must meet the specifications given in Paragraph 1 above within plus or minus 0.5%.

c. Analyses

i. The Vendor must furnish with each lot of each material an ultraviolet spectrum of the material. This spectrum shall cover the wavelength range from 285 to 315 nanometers.

ii. In addition, each lot of liquid propellant must be furnished with the following analytical data: titration with alcoholic butyl amine, and weight-per cent of carbon.

C.4.1.8 REPORTS

a. A final report must be prepared as part of this contract. This report shall contain a complete and detailed description of all of the technical effort expended on this contract. All of the procedures used both for preparation and for analysis must be documented.

b. This report must be prepared in accordance with the Mil Spec governing Department of Defense reports. A copy of this spec shall be made available on request. (Reference DI-S-4057)

c. If this final report contains proprietary information, this fact must be stated to the Contracting Officer's Technical Representative, and the report shall contain appropriate notification of this fact. Any such proprietary information shall be protected from unauthorized release or distribution. If there is no proprietary information in the report, it shall be published as a BRL Contractor Report.

PART I - THE SCHEDULE
SECTION D - PACKAGING AND MARKING

D.5100 PACKAGING:

The liquid propellant shall be shipped as follows:

- a. Only 10-liter or 2.5 gallon polyethylene bottles shall be used.
- b. The polyethylene must be new (so-called "virgin") material, certified for food-service. (Such bottles are readily available from several suppliers)
- c. The closures on the bottles shall have some kind of "tamperproof" closure. It is not the intention to make this closure too tight; rather, the intention is to have positive evidence of tampering after the bottles have left the supplier's control.
- c. The closure should permit leakage of gas if the pressure inside the bottle exceeds 14.7 psi gauge pressure (that is, a pressure 14.7 psi above ambient).

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APPENDIX B:
SHIPPING ARRANGEMENT DRAWING

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INTENTIONALLY LEFT BLANK.

APPENDIX C:
MSDS/IHSC FORM

INTENTIONALLY LEFT BLANK.

REACTIVITY	2	Thiokol CORPORATION TACTICAL OPERATIONS Elkton Division 55 THIOKOL ROAD, ELKTON, MD. 21921-0241 301-392-1000	
	2		
	3		
— HAZARD RATING — 4-EXTREME 3-HIGH 2-MODERATE 1-SLIGHT 0-RELATIVELY HARMLESS		DATE ISSUED 10/12/87	MSDS NO. 111B

LIQUID GUN PROPELLANT WARNING

MASS FIRE HAZARD IF CONFINED

- Do not attempt to extinguish.
- Burning propellant emits toxic gases.
- DoD Hazard Class 1.3C.

CORROSIVE

- Liquid corrosive effect. (slight)
- Mild oxidizing solution.
- Reacts with reducing materials.
- Avoid contact with metals and metal powders.

HEALTH EFFECTS

- Skin and eye irritant/corrosive.
- Avoid inhalation of aerosolized mists/vapors.
- Avoid ingestion and skin absorption of LGP.
- May cause hypotension, methemoglobinemia or HEINZ body formation in blood.
- Experimental material, hazards have not been fully characterized.

E39846-15

Figure C-1. Liquid Gun Propellant Hazards Communication Shipping Label

Morton Thiokol, Inc.
Elkton Division
P.O. Box 241
Elkton, Maryland 21921-0241
Emergency Phone (301) 398-3000

MATERIAL SAFETY DATA SHEET

MSDS NO. 111
Date Issued 10/12/87
Written By R. L. Jenkins
Approved By R. L. Jenkins

I. PRODUCT IDENTIFICATION

A. Trade Name and Synonyms: Liquid Gun Propellant, LGP 1846

II. PHYSICAL DATA

- A. Appearance and Odor: Water white to very pale straw colored solution. Odorless.
B. Volatiles: Water.

III. COMPOSITION

Hazardous Ingredients	Percent	OSHA PEL	ACGIH TLV
A. Hydroxylammonium Nitrate	61	--	NE
B. Triethanolammonium Nitrate	19	--	NE
C. Water	20	--	--

IV. HEALTH HAZARD DATA

- A. Threshold Limit Value: Not established.
B. Effects of Overexposure:
1. Respiratory: None known. Decomposition products are known to cause breathing difficulty and respiratory damage.
2. Eyes: Corrosive liquid effect, moderately severe eye injury if contacted with eyes. Decomposition products can be irritating.
3. Skin: Acidic, pH = 4, corrosive liquid effect, no known dermatitis.
4. Skin Absorption: No absorption observed.
5. Ingestion: Corrosive liquid effect may cause nausea, abdominal discomfort, and collapse. Decomposition products are also corrosive to internal organs.
6. Other: None currently known.

NE = Not Established

V. EMERGENCY AND FIRST-AID PROCEDURES

- A. Inhalation: If decomposition products are inhaled, remove victim to fresh air. Call a physician and/or emergency facility immediately.
- B. Eyes: Immediately flush eyes with large amounts of water for at least 15 minutes. Call a physician and/or emergency facility immediately.
- C. Skin: Contact with the skin should be treated as with any corrosive material. Immediately flush the area with water. Wash with soap and water.
- D. Ingestion: Accidental ingestion requires water intake (2 glasses) and induced vomiting.

VI. FIRE AND EXPLOSION HAZARD DATA

- A. Liquid gun propellant is a DOT Class B (1.3) liquid explosive. (Reference: ARBRL-CR-00454, May 1981.) The material does not have a measureable flash point but does have an autoignition temperature (Setchkin) of 310 degrees C (590 F). No explosion occurs in an open fire test and the liquid survives 48 hours at 75 degrees C (167 F). The liquid does not explode under impact of a 4.4-lb weight at 28 inches in a standard cavity test and also does not yield a positive card gap test at 70 cards.
- B. Explosive Limits: DOT Class B explosive (1.3 hazard symbol).
- C. Extinguishing Media: Do not attempt to fight burning propellant. Water, CO_2 , or foam may be used to restrict spreading of fire after bulk of propellant has burned.
- D. Special Fire Fighting Procedures: Propellant ingredients contain oxidizer and fuel. Do not fight fire. If ignited while contained, thrust created while burning may give this propellant uncontrollable ballistic properties. Fire fighting should be limited to preventing the spread of other fires.
- E. Explosion Hazards: Static discharge, impact, friction, and pinch points between hard surfaces can initiate propellant fires and should be avoided. See VII, B.

VII. REACTIVITY DATA

- A. Mild oxidizing solution capable of reacting with reducing materials.
- B. Contact with metals should be avoided. Transition metal ion contamination promotes decomposition.
- C. Best stored in inert polyethylene, polypropylene, or Teflon containers.

VIII. SPECIAL PRECAUTIONS

A. Protective Measures:

1. Acid-rated gloves and safety glasses should be worn when handling or transferring this solution.
2. Avoid contact with eyes, skin, and clothing.
3. Wash hands (contacted areas) thoroughly after handling.
4. Spills should be diluted with water and flushed to a sanitary sewer system.

IX. ENVIRONMENTAL PROTECTION

- ##### A.
- The considerable solubility of the dissolved salts provides adequate protection against buildup of crystals. The diluted aqueous solution provides nonhazardous waste. Can be burned in an incinerator when diluted.

"To the best of our knowledge the information contained herein is correct. All chemicals may present unknown health hazards and should be used with caution. Although certain hazards are described herein, we cannot guarantee that these are the only hazards which exist. Final determination of suitability of the chemical is the sole responsibility of the user. Users of any chemical should satisfy themselves that the conditions and methods of use assure that the chemical is used safely.

NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESSED OR IMPLIED, OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE OR ANY OTHER NATURE ARE MADE HEREUNDER WITH RESPECT TO THE INFORMATION CONTAINED HEREIN OR THE CHEMICAL TO WHICH THE INFORMATION REFERS. "

THIS MATERIAL IS EXPERIMENTAL AND ANY HAZARDS ASSOCIATED WITH IT HAVE NOT BEEN FULLY CHARACTERIZED. THIS MATERIAL HAS BEEN PROVIDED EXCLUSIVELY FOR TESTING AND EVALUATION IN A LABORATORY ENVIRONMENT BY KNOWLEDGEABLE RESEARCH PERSONNEL.

Morton Thiokol, Inc.
Elkton Division
P.O. Box 241
Elkton, Maryland 21921-0241
Emergency Phone (301) 398-3000

Page 1 of 2

MATERIAL SAFETY DATA SHEET

MSDS No. 054
Date Issued 5/12/86
Reviewed by: T.R. Cessario
T. R. Cessario
Safety Manager

I. Product Identification

A. Trade Name and Synonyms:

Liquid Gun Propellant, LGP 1845

II. Physical Data

A. Appearance and Odor:

Water white to very pale straw colored solution, odorless.

B. Volatiles Water.

III. Composition

Hazardous Ingredients	Percent	PEL	TLV
A. Hydroxylammonium Nitrate	63%	----	Not established.
B. Triethanolammonium Nitrate	20%	----	Not established.
C. Water (H ₂ O)	17%	----	----

IV. Health Hazard Data

A. Threshold Limit Value:

Not established.

B. Effects of Overexposure:

1. Respiratory:

N/A

2. Eyes:

Corrosive liquid effect, moderately severe eye injury if contacted with eyes.

3. Skin:

Acidic pH = 4, corrosive liquid effect, no known dermatitis.

4. Skin Absorption:

No absorption observed.

5. Ingestion:

Corrosive liquid effect may cause nausea, abdominal discomfort and collapse.

6. Other:

None currently known.

V. Emergency and First-Aid Procedures

A. Inhalation:

N/A

B. Eyes:

Immediately flush eyes with large amounts of water for at least fifteen (15) minutes.

C. Skin:

Contact with the skin should be treated as with any corrosive material. Immediately flush the area with water. Wash with soap and water.

D. Other:

Ingestion: accidental ingestion would require water intake (2 glasses) and induced vomiting.

VI. Fire and Explosion Hazard Data

- A. Liquid gun propellant is a DOT class B, liquid explosive. (Reference: ARBRL-CR-00454, May 1981). The material does not have a measureable flash point but does have an autoignition temperature (Setchkin) of 310°C (590°F). No explosion occurs in an open fire test and the liquid survives 48 hours at 75°C (167°F). The liquid does not explode under impact of a 4.4 lb weight at 28 inches in a standard cavity test and also does not yield a positive card gap test at 70 cards.

MATERIAL SAFETY DATA SHEET (CONTINUED)

VII. Reactivity Data

- A. Mild oxidizing solution, capable of reacting with reducing materials.
- B. Contact with metals should be avoided. Transition metal ion contamination promotes decomposition.
- C. Best stored in inert polyethylene, polypropylene or teflon containers.

VIII. Special Precautions

A. Protective Measures

- 1) Acid rated gloves and safety glasses should be worn when handling or transferring this solution.
- 2) Avoid contact with eyes, skin and clothing.
- 3) Wash hands (contacted areas) thoroughly after handling.
- 4) Spills should be diluted with water and flushed to a sanitary sewer system.

IX. Environmental Protection

- A. The considerable solubility of the dissolved salts provides adequate protection against build up of crystals. The diluted aqueous solution provides non-hazardous waste.

MAY 4 1989 13:38

DISPOSITION FORM

For use of this form, see AR 340-15; the proponent agency is TAGO.

REFERENCE OR OFFICE SYMBOL

SMCAR-SF (385-16b)

SUBJECT

Interim Hazard Classification Extension for Liquid
Propellant 1845 and 1846

FROM

DATE

CMT 1

TO

SMCAR-AEE-B
ATTN: Mr. Downs

SMCAR-SF

20 April 1989
Mrs.Z. Luu/rgs/45558

1. Reference:

- a. AMC-R 385-21, 2 January 1987, Determination and Assignment of Ammunition and Explosives Hazard Classification
- b. Army Technical Bulletin (TB) 700-2, 1 September 1982, Department of Defense Explosives Hazard Classification Procedures
- c. DF, SMCAR-SF, 2 March 1987, Policy Regarding Interim and Final Hazard Classification (IHC and FHC) of Ammunition and Explosives (encl 3)
- d. FONCON between Mr. Elliott, SMCAR-SF and Mr. Leveritt, SLCBR-IB-B, 19 April 1989, Subject: SAB

2. The Interim Hazard Classification (IHC) requested in reference d is at encl 1 and is applicable for shipment and storage of either small or in-process R&D quantities.

3. A copy of this IHC and DOT Exemption No. DOT-E-9256 (third revision) (encl 2) must be carried aboard each mode of transportation authorized by DOT Exemption No. DOT-E 9256 (third revision). In addition, a copy of this IHC will be expeditiously furnished by your office, upon receipt of a proper request, to any organization required to store these propellants.

4. Final Hazard Classification (FHC) TAW refs a, b and c is required for full scale production. To obtain FHC, the information contained in encl 3 should be compiled and forwarded through this office, to AMC Field Safety Activity.

5. In addition, to complete the safety data requirement, it will be necessary to obtain the following data for the liquid propellant 1846 & 1845.

Test needed for liquid propellant 1846

- a. Detonation Test
- b. Ignition & Unconfined Burning Test
- c. Explosion Temperature (5 sec)
- d. Friction Test (Apparatus & Comparison Values)

SMCAR-SF

SUBJECT: Interim Hazard Classification for Liquid Propellant 1845 and 1846

Test needed for liquid propellant 1845

- a. Friction Test (Apparatus & Comparison Values)
- b. Explosion Temperature (5 sec)
- c. Electrostatic Discharge Test

3 Encls
As stated

Charles R. Peters
CHARLES R. PETERS
Chief Hazard Classification Branch



DEPARTMENT OF THE ARMY
ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER
PICATINNY ARSENAL NEW JERSEY 07806-5000

REPLY TO
ATTENTION OF

SMCAR-SF (385-16b)

19 April 1989

MEMORANDUM FOR: Commander, Armament Research, Development and Engineering Center, ATTN: SMCAR-AEE-B (Mr. Downs), Picatinny Arsenal, New Jersey 07806-5000

SUBJECT: Interim Hazard Classification No. 219-A-0293

1. Reference: a. Memorandum w/encls, SLCBR-IB-B, 28 Mar 88, Subject: Request for Interim Hazard Classification.
b. Report, BRL, May 1981, Subject: Classification of Liquid Propellant and RAW-Materials for Transportation and Storage.
2. Item: Propellant, Liquid LP 1846 and 1845 f/Large and Medium Caliber guns
3. Reference Document: Ref a
4. Applicable Sections of Title 49 Code of Federal Regulations (49 CFR) for proper description and classification: 173.88(f)
5. Interim Hazard Classification:
 DOD Hazard Class: Group II (Liquid Propellant Storage Hazard Class)
 DOD Storage Compatibility Group: A (Liquid Propellant storage Group)
 DOT Hazard Class: Class B Explosive
 DOT Container Marking: Propellant Explosives (Liquid), Class B
 UN Serial Number: 0190
 Explosive Weight for QD: 60 lbs (27.25 kgs)
6. Period of Applicability: 6 months
7. Expiration Date: 19 Oct 89
8. a. Sections of Title 49 Code of Federal Regulations for Packaging:
 Packaging: Section 173.93(e)
 Marking: Section 172, Subpart D and Section 173.93(f)
 Labeling: Section 172, Subpart E (172.411) Explosive B Label
 b. Packaging Drawings: Dwg No. E40378, Rev-D (Morton Thiokol Inc.)
9. The point of contact is Ziem Luu, SMCAR-SF, (201) 724-5558.

FOR THE COMMANDER:

James E. Elliott
JAMES E. ELLIOTT

Acting Chief, Safety Office

CP:
AMXOS-SE, DDESB-KT



US Department
of Transportation
Research and
Special Programs
Administration

: DOT-E 9256
(THIRD REVISION)

400 Seventh St. S.W.
Washington D.C. 20590

NOV 5 1987

1. U.S. Department of Defense (DOD), Washington, D.C., and its contractors are hereby granted an exemption from certain provisions of the Department's Hazardous Materials Regulations to ship various kinds of explosives in commerce with a DOD Interim Hazard Classification. These shipments are subject to the limitations and special requirements specified herein and this exemption provides no relief from any regulation other than as specifically stated herein.

2. BASIS. This exemption is based on DOD's application dated September 4, 1987, submitted in accordance with 49 CFR 107.105 and the public proceeding thereon.

3. HAZARDOUS MATERIALS (Descriptor and class). Military explosives, explosive devices and ammunition - classed as explosive A, B or C on an interim basis.

4. PROPER SHIPPING NAME (49 CFR 172.101). The proper shipping name or generic description for each explosive, as appropriate.

5. REGULATION AFFECTED. 49 CFR Part 107, Appendix B, Subpart B(1) and 173.86; 173.30; 46 CFR 146.20-13.

6. MODES OF TRANSPORTATION AUTHORIZED. Motor vehicle, rail freight, cargo vessel, cargo aircraft only (see paragraph 8e).

7. SAFETY CONTROL MEASURES.

a. Authorized Explosives. An explosive authorized to be transported under the provisions of this exemption is a new explosive covered by a written DOD Interim Hazard Classification that has been issued not more than one year before the date of transportation of the explosive.

b. Classification of Explosives. Each DOD Interim Hazard Classification of an explosive must be approved (by signature) by one of the following persons:

Chief, Explosive Safety Branch (AFISCI-SEV)
Norton Air Force Base, CA 92409

Director of Safety
Combat Systems Directorate (06H)
Naval Sea Systems Command
Washington, DC 20362

Continuation of 3rd Rev., DOT-E 9256

NOV 5 1987

Page 2

Chief
Safety Engineering Division
DARCOM Field Safety Activity
Charlestown, IN 47111

The names of the persons holding the above listed positions shall be provided to the Office of Hazardous Materials Transportation (OHMT) on a current basis.

c. Delegation of Approval Authority. The chiefs of the U.S. Army and U.S. Air Force offices identified in 49 CFR 173.86(b)(2) may, with the Department of Defense Explosives Safety Board's (DDESB) concurrence, authorize, respectively, the following additional persons to grant (by signature) interim hazard classification approvals, subject to all the conditions and procedures of this exemption:

U.S. Army:

Chief, Safety Office
U.S. Army Missile Command
Redstone Arsenal, AL 25898-5130

Chief, Safety Office
U.S. Army Armament Research and Development Center
Dover, NJ 07801-6001

Chief, Safety Office
U.S. Army Chemical Research and Development Center
Aberdeen Proving Ground, MD 21010-5423

U.S. Air Force:

Chief, Weapons Safety
HQ, Air Force Systems Command
Andrews Air Force Base, MD 20334-5000

Director of Systems Safety
HQ, Armament Division (AFSC)
Eglin Air Force Base, FL 32542-5000

The names of the persons holding the above listed positions to whom approval authority is delegated shall be provided to the OHMT and to DDESB-KT on a current basis.

8. SPECIAL PROVISIONS.

a. This exemption applies only to the transportation of new explosives covered by DOD Interim Hazard Classifications. Also, this exemption authorizes DOD contractors to offer shipments of new explosives, which have been assigned a DOD interim hazard class, under commercial bills of lading.

b. A copy of the DOD Interim Hazard Classifications must be carried aboard each motor vehicle, cargo vessel and aircraft used to transport explosives covered by this exemption.

c. Packages are not required to be marked with the exemption number.

d. The requirements of 49 CFR 173.86 do not apply to explosives transported under this exemption.

e. Shipments by cargo aircraft must be transported by an air carrier authorized by an exemption to carry explosives. The air carrier, exemption held by the carrier, and type of explosive to be carried, must be identified to and approved in writing by this Office prior to the first shipment.

9. REPORTING REQUIREMENTS. Any incident involving loss of contents of the package must be reported to the OHMT as soon as practicable.

10. EXPIRATION DATE. October 31, 1989.

Issued at Washington, D.C.:



Alan I. Roberts
Director
Office of Hazardous Materials Transportation

NOV 5 1987

(DATE)

Address all inquiries to: Director, Office of Hazardous Materials Transportation, Research and Special Programs Administration, U.S. Department of Transportation, Washington, D.C. 20590. Attention: Exemptions Branch.

Dist: PHWA, FRA, FAA, USCG

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APPENDIX D:
REPRESENTATIVE QUALITY LAB DATA

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LABORATORY REPORT


SOUTHWESTERN ANALYTICAL CHEMICALS INC

TO: Morton Thiokol
55 Thiokol Road
Elkton, MD 21921

DATE: 04/08/88

SAMPLE:

Lab No:

Date Submitted:

Description:

Hydroxylamine Nitrate, 2.8 Molar,
Aqueous Solution, Catalog #417
Your PO #24712 WM, Partial Release #4

RESULTS:

Lot #8098Customer Specifications

Assay, %	24.76	25.0 ± 1.0
SO ₄ (sulfate), ppm	< 10	10 max
Cl (chloride), ppm	1.6	10 max.
Ash, ppm	< 10	10 max.
Stabilizer(Free HNO ₃)	0.008 Molar	0.01 Molar max.
Total Ca plus Mg, ppm	< 2	10 max.
Ba (barium), ppm	< 0.1	0.1 max.
Fe (iron), ppm	0.04	0.2 max.
Al (aluminum), ppm	< 0.1	0.2 max.
Pb (lead), ppm	< 0.1	
Ni (nickel), ppm	< 0.01	
Sn (tin), ppm	< 0.01	
Cu (copper), ppm	< 0.01	
Mg (magnesium), ppm	0.13	
Mn (manganese), ppm	< 0.01	

REMARKS:

By 

INTER-OFFICE MEMO
CONTROL LABORATORY

REF: QAL/139-89
DATE: 3 October 1989

To: R. G. Brasfield

CC: J. Mallick, R. R. Weldin, R. A. Biddle, J. L. Seador,
J. J. Lehman, File

FROM: L. S. White

SUBJECT: TEAN Analysis

TEAN Sublots 89-030-1,2,3,4, 89-031-1,2,3,4, 89-032-1,2,3,4

TEAN, % (titration)	79.36
Moisture, %	20.33
UV, %	79.53

TEAN Sublots 89-033-1,2,3,4, 89-034-1,2,3,4, 89-035-1,2,3,4

TEAN, % (titration)	79.56
Moisture, %	19.90
UV, %	81.08

L.S. White
L.S. White
10/3/89
Date

INTER-OFFICE MEMO
Quality Laboratory

Ref: QAL/117-89
Date: 9 August 198

TO: R. G. Brasfield
CC: J. Mallick, R. R. Weldin, R. A. Biddle, J. L. Seador,
File
FROM: S. A. Bonsell
SUBJECT: HAN Analysis

HAN Lot 89-008

	<u>% HAN (titration)</u>	<u>% Moisture</u>
Drum 1	81.4	18.5
Drum 2	81.3	18.2
Drum 3	81.3	18.1
Drum 4	81.1	18.3
Drum 5	81.1	18.0
Drum 6	81.0	18.6

Material is released for further processing.

Quiley A Bonsell LAB SUPERVISOR

8/9/89 DATE

SAB/asm

INTER-OFFICE MEMO
Quality Laboratory

Ref: QAL/146-89
Date: 5 October 1989

TO: R. G. Brasfield

CC: J. Mallick, R. B. Weldin, R. A. Biddle,
W. R. Brownell, J. J. Lehman, File

FROM: S. A. Bonsell

SUBJECT: LGP 1846-09 Analysis

LOT 1846-09

	Sample 1	Sample 2	Sample 3
MOISTURE, % (KF)	19.5	19.5	19.8
HAN, %	60.1	60.1	60.0
TEAN, %	20.7	20.7	20.6

Material is released for further processing.

Shirley A. Bonsell Lab Supervisor
10/5/89 Date

INTER-OFFICE MEMO
Quality Laboratory

Ref: QAL/147-89
Date: 9 October 1989

TO: R. G. Brasfield

CC: J. Mallick, R. R. Weldin, R. A. Biddle,
W. R. Brownell, J. J. Lehman, File

FROM: S. A. Bonsell *SAB*

SUBJECT: LCP 1846-09 Analysis

	LOT 1846-09 First Adjustment		
	Sample 1	Sample 2	Sample 3
MOISTURE, % (KF)	20.30	20.20	20.29
HAN, %	60.04	60.07	60.19
TEAN, %	19.37	19.45	19.41

Material is released for further processing.

S. A. Bonsell Lab Supervisor
10/9/89 Date

INTER-OFFICE MEMO
Quality Laboratory

Ref: QAL/150-89
Date: 12 October 1989

TO: R. G. Brasfield

CC: J. Mallick, R. R. Weldin, R. A. Biddle,
W. R. Brownell, J. J. Lehman, File

FROM: S. A. Bonsell

SUBJECT: LGP 1846-09 Analysis

LOT 1846-09

MOISTURE, % (KF)	20.4
HAN, %	61.1
TEAN, %	19.3

Material is released for packaging.

SA Bonsell

Lab Supervisor

10/12/89

Date

INTER-OFFICE MEMO
Quality Laboratory

Ref: QAL/3-90
Date: 05 January 1990

TO: R. G. Brasfield
CC: J. Mallick, R. R. Weldin, R. A. Biddle, W. R. Brownell,
J. J. Lehman, File
FROM: S. A. Bonsell
SUBJECT: LGP 1846-09 Analysis

Lot 1846-09

	Results -----	Limits -----
MOISTURE, % (KF)	20.4	19.5 - 20.5
HAN, % (Naoh titration)	61.1	60.3 - 61.3
TEAN, % (by difference)	19.3	18.7 - 19.7
NITRATE, moles (100g)	trace attached	info

Trace Metals:

Fe (ppm)	2	<5
Ni (ppm)	<1	<5
Cu (ppm)	<1	<5
Pb (ppm)	<1	<5
Sn (ppm)	<1	<5

Material is released for shipment.

S. A. Bonsell
1/5/90

Lab Supervisor

Date

10/16/89

64P1846-9-59

10/16/89

64P1846-9-28

10/16/89

64P1846-9-1

APPENDIX E:
ANALYTICAL PROCEDURES ANALYSIS WORKSHEETS
PRODUCT RELEASE FORMS

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ANALYTICAL PROCEDURES FOR LGP AND COMPONENTS

The attached procedures for the analysis of the in-process components and final mixtures of LGP compositions based on the TEAN - HAN - H₂O system are written for experienced personnel and are not considered appropriate for direct use without interpretation by a trained Chemist. No specification is made as to the instrumentation required. These procedures can be adapted to most automated titrimeters or to manual titration. Either a constant wavelength or a recording UV spectrophotometer can be used for the total nitrate concentration determination. The recording spectrophotometer is only required when a spectrogram is specified in the delivery of the final product LGP. Trace metal contamination analyses can be conducted using either AA or ICP instrumentation. The AA technique is adequate for acceptance of the LGP.

Analysis worksheets and components or product release sheets are also included with these procedures to aid in the application of the procedures.

TRIETHANOLAMMONIUM NITRATE (TEAN) SOLUTION

1. Titration With 0.2XXX N Aqueous Sodium Hydroxide

- 1a. Pipette 5.0 ml of sample into a preweighed 30 ml beaker and reweigh to the closest 0.01mg (enter the weights and calculate the density in g/ml on the Data Sheet). Transfer all of the contents of the beaker, washing thoroughly, into a 500 ml Volumetric flask. Dilute to the mark with demineralized water (enter the values for dilution on the Data Sheet).
- 1b. Pipette 50.0 ml aliquot of solution into a 150 ml beaker and add 50.0 ml of demineralized water. Titrate to the end-point with 0.2XXX N NaOH. Enter the value for the end-point and calculate the % TEAN on the Data Sheet. Titration should be run in triplicate.

2. Nitrate Determination by UV

- 2a. Prepare and standardize a 0.1350 ± 0.0010 N HNO_3 standard using 0.2XXX N NaOH.
- 2b. Determine the absorbance of the HNO_3 standard at 302 nanometers using the appropriate instrument settings. Enter the absorbance and calculate the moles NO_3/Abs on the Data Sheet.
- 2c. Determine the absorbance of the TEAN solution prepared in 1a. Enter the absorbance values and calculate the % TEAN on the Data Sheet.

3. Water Content by Karl Fischer Titration

- 3a. Fill a microsyringe with TEAN solution and weigh to the closest 0.01mg. Add 6-9 drops of the solution into the predried carrier solution and titrate to the endpoint using a Karl Fischer Aquameter. Reweigh the microsyringe as soon as possible after introduction of the sample. Using the volume (V, ml) of the titrant, the titer of the titrant (T, mg $\text{H}_2\text{O}/\text{ml}$) and the sample weight (W, g), calculate the % H_2O .

$$\% \text{H}_2\text{O} = \frac{10 VT}{W}$$

The titration should be run in duplicate.

HYDROXYLAMMONIUM NITRATE (HAN) SOLUTION ($\approx 24\%$) AS RECEIVED

4. Titration With 0.2XXX N Aqueous Sodium Hydroxide

- 4a. Pipette 20.0 ml of sample into a preweighed 30 ml beaker and reweigh to the closest 0.1mg (enter the weights and calculate the density in g/ml on the Data Sheet). Transfer all of the contents of the beaker, washing thoroughly, into a 500 ml Volumetric flask. Dilute to the mark with demineralized water (enter the values for the dilution on the Data Sheet).
- 4b. Pipette 20.0 ml aliquot of solution into a 150 ml beaker and dilute with 80 ml of demineralized water. Titrate to the end-point with 0.2XXX N NaOH. Enter the values for the end-point of the titration and calculate the % HAN on the Data Sheet. Titration should be run in triplicate.

5. Nitrate Determination By UV

- 5a. Prepare and standardize a 0.1350 ± 0.0010 N HNO_3 standard using 0.2XXX N NaOH.
- 5b. Determine the absorbance of the HNO_3 standard at 302 nanometers using the appropriate instrument settings. Enter the absorbance and calculate the moles NO_3/Abs on the Data Sheet.
- 5c. Determine the absorbance of the HAN solution prepared in 4a. Enter the values and calculate the % HAN on the Data Sheet.

HYDROXYLAMMONIUM NITRATE (HAN) SOLUTION
(≈84%) AS CONCENTRATED

6. Titration With 0.2XXX N Aqueous Sodium Hydroxide

- 6a. Pipette 5.0 ml of sample into a preweighed 30 ml beaker and reweigh to the closest 0.01mg (enter the weights and calculate the density in g/ml on the Data Sheet). Transfer all of the contents of the beaker, washing thoroughly, to a 500 ml volumetric flask. Dilute to the mark with demineralized water (enter the values for the dilution on the Data Sheet).
- 6b. Pipette 25.0 ml aliquot of solution into a 150 ml beaker and dilute with 75 ml of demineralized water. Titrate to the end-point with 0.2XXX N NaOH (enter the values for the end-point of the titration on the Data Sheet). Titration should be run in triplicate.

7. Nitrate Determination by UV

- 7a. Prepare and standardize a 0.1350 ± 0.0010 N HNO_3 standard using 0.2XXX N NaOH.
- 7b. Determine the absorbance of the HNO_3 standard at 302 nanometers using the appropriate instrument settings. Enter the absorbance and calculate the moles NO_3/Abs on the Data Sheet.
- 7c. Determine the absorbance of the HAN solution prepared in 6a. Enter the values and calculate the % HAN on the Data Sheet.

8. Water Content by Karl Fischer Titration

- 8a. Fill a microsyringe with HAN solution and weigh to the closest 0.01mg. Add 6-9 drops of the solution into the predried carrier solution and titrate to the end-point using a Karl Fischer Aquameter. Reweigh the microsyringe as soon as possible after introduction of the sample. Using the volume (V, ml) of the titrant, the titer of the titrant (T, mg $\text{H}_2\text{O}/\text{ml}$) and the sample weight (W, g), calculate the % H_2O .

$$\% \text{H}_2\text{O} = \frac{10 \text{ VT}}{W}$$

The titration should be run in duplicate.

LIQUID GUN PROPELLANT
LGP1845 AND LGP1846

9. Titration With Aqueous 0.2XXX N Sodium Hydroxide

- 9a. Pipette 4.0 ml of sample into a preweighed 100 ml Volumetric flask and reweigh to the closest 0.1 mg (enter the weights and calculate the density in g/ml on the Data Sheet). Dilute to volume with demineralized water.
- 9b. Pipette 9.0 ml aliquot of solution into a 100 ml beaker, add 50 ml of methanol, add 2.0 ml of Acetone and warm the beaker to $\approx 40^{\circ}\text{C}$ for about 1 minute. Titrate to a double break end-point with 0.2XXX N NaOH. Enter the values of the end-points and calculate the % HAN and the % TEAN on the Data Sheet. Titration should be run in triplicate.

10. Total Nitrate Determination by UV

- 10a. Prepare and standardize a 0.1350 ± 0.0010 N HNO_3 standard using 0.2XXX N NaOH.
- 10b. Determine the absorbance of the HNO_3 standard at 302 nanometers using the appropriate instrument settings. Enter the absorbance and calculate the moles NO_3/Abs on the Data Sheet.
- 10c. Pipette 5.0 ml of LGP sample into a preweighed 30 ml beaker and reweigh to the closest 0.01 mg. Transfer all of the contents of the beaker, washing thoroughly, into a 500 ml volumetric flask. Dilute to volume with demineralized water.
- 10d. Determine the absorbance of the above solution. Enter the values and calculate the total moles of nitrate per 100 g on the Data Sheet.

11. Water Content By Karl Fischer Titration

- 11a. Fill a microsyringe with LGP solution and weigh to the closest 0.01 mg. Add 6-9 drops of the solution into the predried carrier solution and titrate to the end-point using a Karl Fischer Aquameter. Reweigh the microsyringe as soon as possible after introduction of the sample. Enter the values and calculate the % H_2O on the Data Sheet. The titration should be run in triplicate.

FREE ACID DETERMINATION IN HAN OR LGP SOLUTION

12. Acid Titration

- 12a. Pipette 50.0 ml of solution into a 150 ml beaker and add 50 ml of demineralized H₂O.
- 12b. Set pH on titrimeter to the 0-5 pH full scale range.
- 12c. Titrate solution with 0.2XXX N NaOH and determine value of titrant to end-point (\approx 2.5 pH).
- 12d. Calculate free acid by the following equation:

$$\frac{(V_b) (M_b)}{(V_s)} = M_s$$

Where: V_b = Volume base

V_s = Volume Solution (50 ml)

M_b = Molarity of base (0.2XXX)

M_s = Molarity of Free Acid in Solution

TRACE METAL CONTAMINATION ANALYSIS

13. Determination of Trace Metals

- 13a. Prepare appropriate concentration of metal standard solutions (e.g., 1 ppm Fe, Ni, Cu, and Pb and 50 ppm Sn for AA technique).
- 13b. Prepare solutions for analysis by weighing 30.0 ± 0.1 grams of sample to be analyzed into a 100 ml volumetric flask and dilute to mark with demineralized water.
- 13c. Set appropriate instrument parameters for each element to be analyzed and obtain the absorbance of both the standard and the sample solutions.
- 13d. Calculate the ppm of each element from the calibration curve obtained for each standard solution.

HAN - TEAN

SAMPLE _____

DATE _____

WEIGHTS

_____ g. Wt. S + B

_____ g. Wt. Beaker

_____ g. Wt. Sample

_____ g W_s = _____ $\frac{g}{ml}$ Density

_____ ml $V_{Pipette}$

DILUTION

_____ g W_s = _____ $\frac{g}{ml}$ $\frac{W_s}{V_F}$

_____ ml V_{Flask}

TITRATION

_____ $\frac{g}{ml}$ $\frac{W_s}{V_F}$ x 1000 $\frac{mg}{g}$ x _____ ml V_P = _____ mg W_s

$$1. \quad \frac{\text{_____ } ML_b \times \text{_____ } N_b \times \text{_____ } M_w \times 100}{\text{_____ } mg \ W_s} = \text{_____ } \%$$

$$2. \quad \frac{\text{_____ } ML_b \times \text{_____ } N_b \times \text{_____ } M_w \times 100}{\text{_____ } mg \ W_s} = \text{_____ } \%$$

$$3. \quad \frac{\text{_____ } ML_b \times \text{_____ } N_b \times \text{_____ } M_w \times 100}{\text{_____ } mg \ W_s} = \text{_____ } \%$$

NO₃ UV

_____ Mol HNO₃ = _____ $\frac{\text{Mol NO}_3}{\text{Abs}}$
_____ Abs CAL

_____ Abs S x _____ Mol NO₃ _____ MW = _____ %

_____ $\frac{g}{ml}$ $\frac{W_s}{V_F}$ x 10

HAN MW = 96.04, TEAN MW = 212.20

SAMPLE _____

DATE _____

_____ g W_s = _____ g/ml Density_____ ml V_{Pipette} _____ W_s gms/100 ml_____ gms/ml x _____ ml = _____ gms x 1000 mg/gm = _____ mg W_s _____ V_o _____ V_o _____ V_o Break_____ V_{1st} _____ V_{1st} _____ V_{1st} Break_____ V_{2nd} _____ V_{2nd} _____ V_{2nd} Break_____ V_{2nd} Ave. _____ N x 100 = _____ Moles $\frac{NO_3}{100g}$

$$\% \text{ HAN} = \frac{(V_{1st} - V_o) \text{ ml} \times N \times 96.04 \times 100}{W_s} \quad \text{_____ N NaOH}$$

$$\% \text{ HAN} \text{ _____} = \frac{(\text{_____} - \text{_____}) (\text{_____ N}) (96.04) (100)}{\text{_____ mg } W_s}$$

$$\% \text{ HAN} \text{ _____} = \frac{(\text{_____} - \text{_____}) (\text{_____ N}) (96.04) (100)}{\text{_____ mg } W_s}$$

$$\% \text{ HAN} \text{ _____} = \frac{(\text{_____} - \text{_____}) (\text{_____ N}) (96.04) (100)}{\text{_____ mg } W_s}$$

$$\% \text{ TEAN} = \frac{(V_{2\text{nd}} - V_{1\text{st}}) \text{ ml} \times N \times 212.2 \times 100}{\text{mg } W_s}$$

$$\% \text{ TEAN} \quad \underline{\hspace{2cm}} = \frac{(\underline{\hspace{2cm}} - \underline{\hspace{2cm}}) (\underline{\hspace{2cm}} N) (212.2) (100)}{\underline{\hspace{2cm}} \text{ mg } W_s}$$

$$\% \text{ TEAN} \quad \underline{\hspace{2cm}} = \frac{(\underline{\hspace{2cm}} - \underline{\hspace{2cm}}) (\underline{\hspace{2cm}} N) (212.2) (100)}{\underline{\hspace{2cm}} \text{ mg } W_s}$$

$$\% \text{ TEAN} \quad \underline{\hspace{2cm}} = \frac{(\underline{\hspace{2cm}} - \underline{\hspace{2cm}}) (\underline{\hspace{2cm}} N) (212.2) (100)}{\underline{\hspace{2cm}} \text{ mg } W_s}$$

KARL FISCHER % H₂O

$$\underline{\hspace{2cm}} \frac{(\underline{\hspace{2cm}} \text{ ml KF}) (\underline{\hspace{2cm}} \frac{\text{mg H}_2\text{O}}{\text{ml KF}})}{W_s \text{ gm} \times 10} = \underline{\hspace{2cm}} \% \text{ H}_2\text{O}$$

 $W_s \text{ gms}$

$$\underline{\hspace{2cm}} \frac{(\underline{\hspace{2cm}} \text{ ml KF}) (\underline{\hspace{2cm}} \text{ ml KF})}{W_s \text{ gm} \times 10} = \underline{\hspace{2cm}} \% \text{ H}_2\text{O}$$

 $W_s \text{ gms}$

$$\underline{\hspace{2cm}} \frac{(\underline{\hspace{2cm}} \text{ ml KF}) (\underline{\hspace{2cm}} \text{ ml KF})}{W_s \text{ gm} \times 10} = \underline{\hspace{2cm}} \% \text{ H}_2\text{O}$$

 $W_s \text{ gms}$

TEAN

SAMPLE IDENTIFICATION: _____

COMMENTS: _____

ANALYSES

1) Solution Density: Hydrometer _____ g/cc Temperature _____ °

Pipette _____ g/cc

2) TEAN Content by Titration _____ %

3) Nitrate Content (UV) as TEAN _____ %

4) H₂O Content (KF) _____ %

5) Trace Metals: Fe _____ ppm

Ni _____ ppm

Cu _____ ppm

Pb _____ ppm

Sn _____ ppm

Material Approved for: _____

Released by: _____

Date: _____

DILUTE OR CONCENTRATED HAN SOLUTION

SAMPLE IDENTIFICATION: _____

COMMENTS: _____

ANALYSES

1) Density Hydrometer _____ g/cc Temperature _____ °

Pipette _____ g/cc

2) HAN Content by Titration _____ %

3) Nitrate Content (UV) as HAN _____ %

4) Water Content (KF) _____ %

5) Free Acid Content _____ M

6) Trace Metals: Fe _____ ppm

 Ni _____ ppm

 Cu _____ ppm

 Pb _____ ppm

 Sn _____ ppm

Material Approved for: _____

Released by: _____

Date: _____

LGP 1845

LOT IDENTIFICATION: _____

COMMENTS: _____

ANALYSES

- 1) Density: Hydrometer _____ g/cc
Pipette _____ g/cc
- 2) H₂O Content (KF) _____ % vs 16.3% (min), 17.3% (max)
- 3) Nitrate Content (UV) _____ moles/100g vs 0.7449 (min)
0.7600 (max)
- 4) Oxime/Acid Titration _____ %HAN vs 62.7 (min), 63.7% (max)
_____ %TEAN vs 19.5 (min), 20.5% (max)
- 5) Ratio HAN/TEAN _____ vs 3.06 (min), 3.28 (max)
- 6) Trace metals Fe _____ ppm (<5ppm)
 Ni _____ ppm (<5ppm)
 Cu _____ ppm (<5ppm)
 Pb _____ ppm (<5ppm)
 Sn _____ ppm (<5ppm)

Material Approved for: _____

Released by: _____

Date: _____

LGP 1846

LOT IDENTIFICATION: _____

COMMENTS: _____

ANALYSES

1) Density:	Hydrometer	_____	g/cc
	Pipette	_____	g/cc
2) H ₂ O Content (KF)	_____	% vs 19.5 % (min), 20.5% (max)	
3) Nitrate Content (UV)	_____	moles/100g vs 0.7184 (min)	
4) Oxime/Acid Titration	_____	% HAN vs 60.3 (min), 61.3% (max)	
	_____	% TEAN vs 18.7 (min), 19.7% (max)	
5) Ratio HAN/TEAN	_____	vs 3.06 (min), 3.28 (max)	
6) Trace metals	Fe	_____	ppm (<5ppm)
	Ni	_____	ppm (<5ppm)
	Cu	_____	ppm (<5ppm)
	Pb	_____	ppm (<5ppm)
	Sn	_____	ppm (<5ppm)

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